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RESEARCH AND DEVELOPMENT ON ENHANCED DEGRADABLE PLASTICS

by

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Blends of two plastics, polyethylene and polycaprolactone (PCL) (where the latter is biodegradable in soil), performed well under marine exposure. Films of blends with up to 10 percent of PCL had acceptable physical strength and moderate biodegradability,

Further studies based on the blends are underway. Work is also currently underway on the suitability of water-soluble films, especially those which might be acceptable under MARPOL Annex V restrictions, for heavy-duty plastic bag applications.

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Table of Contents

Section	<u>on</u>
Abstr	act
1.0	Introduction
1.1	Plastics at Sea
1.2	Ecological Concerns
1.3	Statutory History
1.4	Scope of Report
2.0	Development of Definitions
3.0	Experimental Methodology
3.1	Sample Preparation
	3.1.1 Materials
	3.1.2 Processing
3.2	Exposure of Samples to Marine Environment
3.3	Determination of Tensile Properties
3.4	Gel Permeation Chromatography
4.0	Experimental Studies on Assessment of Enhanced Degradability
4.1	Studies on Plastic-Starch Systems
	4.1.1 Detarioration of Starch-Containing Single-Layer Plastic Films Under Marine Exposure Conditions
	4.1.2 Deterioration of Multilayer Plastic Films Containing Starch (and Ott.) Degradable Materials) Under Marine Exposure Conditions
4.2	Blends of Commodity Plastics with Readily Biodegradable Synthetic Polymers
	4.2.1 Deterioration of Polyethylene-Polycaprolactone Blends Under Maine Exposure Conditions
	4.2.2 Characterization of Polycaprolactone Molecula, Weight Loss by Gel Permeation Chromatography
4.3	Other Miscellaneous Polymers
4.4	Design of Accelerated Biodegradation Equipment
5.0	Conclusions and Directions for Further Work
5.1	Conclusions

Table of Contents (continued)

5.2	Directions for Further Work	4 4
6.0	References	4 €
Appe	ndices	
	Appendix A - C Series Tensile Data	
	Appendix B - A Series Tensile Data	
	Appendix C - A Series Unexposed Tensile Data	
	Appendix D - B Series Tensile Data	
	Appendix E - Polypropylene/Polycaprolactone Tensile Data	
	List of Tables	
	Page .	
Num		
1.1	Estimated Quantities of Waste Generated by Different Types of Vessels	
3.1	A Summary of Extrusion Blowing Parameters Used	1 1
4.1	Physical Properties of LLDPE Films (2.1 to 2.2 mil) with Various Amounts of Starch Additive	1 7
4.2	Biodeterioration of Starch-Polymer Single Layer Films Under Marine Exposure Conditions	1 9
4.3	Composition of Three-Layered (A-B-A) Films Containing Biodegradable Additives	
4.4	Multilayer Films - Tensile Data Summary	23
4.5	Multilayer Films - Ultimate Elongation Percent Decrease	24
4.6	Selected Properties of Polycaprolactone P-700	3 ·
4.7	Polyethylene/Polycaprolactone Films - Tensile Data Summary	33
4.8	Polyethylene/Polycaprolactone Films - Ultimate Elongation Percent Decrease	3 5
4.9	Durations of Marine Exposure Carried Out During Reporting Period	38
	List of Figures	
3.1	Site of Marine Exposure	1 2
3.2	Frame Assembly with Film Samples	1 3
3.3	Exposure Frame with Film Samples	14

List of Figures (continued)

Numb	<u>per</u>	<u>Page</u>
4.1	Percent Ultimate Elongation Retained After Six Months of Exposure for Single Layer Films	_ 20
4.2	Multilayer Films - Percent Decrease in Ultimate Elongation	_ 27
4.3	Biofouling of Films Exposed Under Marine Conditions	_ 30
4.4	Polyethylene/Polycaprolactone Films - Percent Decrease in Ultimate Elongation	_ 36
4.5	Comparison of Percent Ultimate Elongation Retained After Six Months of Exposure for Synthetic Polymer Blends and Starch-Filled Single Layer Films	e _ 37
4.6	GPC Curves for Selected PE/PCL Films	_ 39
4.7	Schematic Diagram of Accelerated Biodegradation Equipment	42
4.8	Accelerated Biodegradation Equipment	_ 43

ABSTRACT

Three major activities carried out during the report period are those relating to (a) development of definitions, standards and test protocols for enhanced biodegradable plastics, (b) the testing of selected starch-based plastic films for biodegradation under marine conditions, and (c) the assessment of two approaches to improved biodegradability in plastic films.

Through presentations and publication in the ASTM journal, an effort was initiated within the American Society for Testing and Materials (ASTM) to develop definitions and test methods. The project's involvement ensures that the Navy requirements will not be compromised in the process.

Commercial starch-containing plastic films degrade slower at sea than that claimed for soil exposure. Moderate rates of degradation of greater than 9 to 12 months require high levels of starch, thus making it difficult to design a film thick enough for heavy-duty bags, yet retaining moderate biodegradability.

Coextrusion of films with the starch (or other degradable material) in the middle layer coated on either side with unfilled plastic material may have improved surface and other properties. These too, however, degrade at rates comparable to or slower than the commercial materials.

Blends of two plastics, polyethylene and polycaprolactone (PCL) (where the latter is biodegradable in soil), performed well under marine exposure. Films of blends with up to 10 percent of PCL had acceptable physical strength and moderate biodegradability.

Further studies based on the blends are underway. Work is also currently under way on the suitability of water-soluble films, especially those which might be acceptable under MARPOL Annex V restrictions, for heavy-duty plastic bag applications.

STUDIES ON DISPOSABLE PLASTIC FILMS FOR SHIPBOARD WASTE DISPOSAL

1.0 Introduction

In spite of their short history, synthetic polymers, particularly plastics, have gained wide popularity as the material of choice in a wide range of packaging, building, and other applications. With the current consumption of plastics reaching around 50 billion pounds in the United States [Modern Plastics, 1988], plastics will without doubt continue to replace conventional materials such as glass, metal, wood, and paper in a variety of additional uses. The projected production in the year 2000 is expected to be in the region of 75 to 100 billion pounds.

The popularity of plastics in packaging and other applications is attributed to the unique characteristics of the material. These include light weight, excellent mechanical strength (tensile properties, tear resistance, impact resistance, etc.), readily controllable and superior optical properties (clarity, gloss, color, etc.), biological inertness, easy processability, low cost, and outstanding durability. Because they are synthetic materials, plastic compositions might be "tailor-made," within limits, to obtain specific useful characteristics. As a response to an historically consistent consumer demand for stronger and longer-lasting plastics, the industry has continually improved the durability of plastics, especially for outdoor exposure conditions. State-of-the-art plastics technology allows materials to be designed so they can satisfactorily function under even the most extreme environmental conditions.

The municipal waste stream in urban areas now consists of about 7 percent post-consumer plastics, a figure that may increase to more than 9 percent by the year 2000 [Franklin Associates, 1988]. While accurate estimates of their lifetimes in the environment are not known, plastics are perceived as being exceptionally persistent materials requiring hundreds of years of exposure to facilitate biodegradation. As most municipal solid waste in the Nation is currently landfilled, the persistence of the plastic waste over extended periods of time is believed to delay the natural "recycling" of landfill volume.

1.1 Plastics at Sea

The recent surge in the use of plastics is also reflected in uses of plastics at sea, particularly by the fishing industry. The most significant source of Marine Pastic debris is gear-related. In addition, passenger, freight, military, and research vessels, as well as beach users introduce plastic materials into the marine environment. Invariably, most

of the nongear-related plastic waste is discharged into the ocean as postconsumer waste from vessels or is washed into the ocean from the beach environment. The magnitude and the nature of this influx of plastics into the sea from such sources vary widely depending on the season of the year and the geographic region.

While the lifetimes of common packaging plastics at sea are not reliably known, it is known that they degrade slowly in the marine environment. Recent experiments have shown that polyethylene film, polypropylene tape, and latex rubber have much slower rates of degradation in seawater than on land [Andrady, 1988a]. Retardation of the degradation process is believed to be due to the lack of "heat buildup" in the samples floating in seawater. Consequently, the samples exposed at sea experienced lower temperatures than those exposed on land.

(a) Fishing Vessels

Introduction of plastics into the world's oceans started in the late 1940's with the changeover from natural fibers (jute, cotton, hemp, etc.) to synthetic polymer fibers in the construction of fishing gear [Uchida, 1985]. Today, nearly all the fishing gear used in developed countries is manufactured from durable synthetic materials, and the commercial fishing industry is the prime source of plastics in the oceans.

A fraction of the gear is invariably lost during active commercial fishing operations and forms a significant percentage of the plastic debris at sea. Of the various types of gear, gill nets, trawls, and traps/pots are considered particularly vulnerable to losses. While reliable quantification of lost gear is difficult, there is little doubt that the losses are considerable [Pruter, 1987; Parker et al. 1987]. In addition to these incidental losses, a significant quantity of netting, ropes, floats, etc., is discarded at sea when damaged gear is repaired.

With a total annual world catch of 82 million tons of fish [Parker et al., 1987], a substantial amount of plastic gear is routinely introduced into the ocean. The estimates of worldwide losses of commercial gear vary from a low of about 1,350 tons annually [NAS, 1975] to as much as 135,000 tons/year [Merrel, 1980]. In addition to gear losses, fishing vessels also discharge "domestic" plastic waste. A 1986 estimate places the number of commercial vessels operating annually in the United States at 125,700 [Parker and Yang, 1986]. The world's fleet of fishing vessels is believed to discharge 23,000 tons of plastics annually into the sea [Horseman, 1985].

(b) Shipping and Transportation Vessels

Shipping and transportation vesels include oil tankers, bulk carriers, and general cargo/passenger liners. With the exception of passenger liners, these vessels carry small crews and are therefore not capable of generating significant quantities of postconsumer

plastic week. The passenger vessels, often carrying as many as a thousand passengers and crew, represent a significant source of plastic waste.

(c) Military Vessels

Military vessels have large crews (the world's fleet carried approximately 2.75 million personnel in 1985) and tend to have voyages of moderate to long durations. Such vessels are likely to produce substantial quantities of waste, including plastic and rubber materials. In addition to the "domestic" consumer plastic waste, the normal operation of the vessel might also generate specialized packaging waste. This latter plastic waste can often represent a significant fraction of the total waste generated on board.

Table 1.1 gives estimates for several categories of vessels [Parker et al., 1987]. Efforts are currently under way to study the composition of shipboard plastics waste.

TABLE 1.1. Estimated Quantities of Waste Generates by Different Types of Vessels.

Vessel Tune	Wast	te Genera	ited	Units
Vessel Type	Plastic	<u>Rubber</u>	Paper	Omes
Fishing Vessels*				
Deep Sea Coastal	42 6	42 6	3749 576	lbs/year/vessels
Shipping & Transportation*				
Crude oil tanker Dry bulk Carrier Container ship	11 9 7	11 9 7	972 783 632	lbs/voyage/vessel
Research Vessel*				
20 - 50 grt >1500 grt	0.08 0.86	0.08 0.86	7.2 77.4	lbs/day/grt range
U.S. Naval Vessels**				
Aircraft Carrier Destroyer Tender Frigate	1050 60 250 40	50 3 12 2	5,500 330 1,300 220	lbs/day

^{*} Data based on Parker et al., 1987.

^{**} Unpublished data from DTRC plastic waste reduction demonstration studies, 1987.

1.2 Ecological Concerns

A detailed discussion of the ecological concerns related to plastic debris at sea is beyond the scope of this report. Several excellent reviews on the fate of the plastic debris, the specific hazards posed by such debris in specific marine species, and the general impact of plastics on the population of target species have been published (Laist, 1987; CEE, 1987; Day et al.,1985).

Available evidence indicates entanglement by the debris and the ingestion of the debris to be the primary concerns with a variety of affected marine animals (including birds, turtles, marine mammals, and fish). These affected populations seem to seek out the debris (either mistaking it as prey or because of mere curiosity); such behavior leads to more fatalities than might be expected on the basis of random encounters with debris. The invariable association of either entangled fish or residual food in most of the plastic waste discharged into the sea also concentrates these species in the same geographic locations that have high incidences of plastic waste. In extreme cases such as with the Hawaiian Monk Seal, recent declines in the natural populations by 4 to 8 %/year have been attributed, at least in part, to entanglement in plastic waste [Fowler, 1985 and 1987].

While reliable estimates of the magnitude of the problem are lacking, there is little doubt that it is serious enough to deserve immediate corrective action.

1.3 Statutory History

The ecological consequences of the occurrence of plastic debris at sea were first brought to public attention by The Standing Committee of the North Pacific Fur Seal Commission in 1970. Studies on the issue indicated that the entanglement hazard posed by derelict netting was a primary cause of the decline in the population of fur seals. To assess the situation and to adopt a strategy to address the problem, the U.S. National Marine and Fisheries Service held a workshop in Hawaii in 1984. Two main recommendations of the workshop on strategies to contain the problem were:

- Education of the public and users of the marine resource, and
- Study of the use of enhanced degradable plastics.

In response, the feasibility of using enhanced degradable plastics in packaging and gear applications was carried out in 1986 (Andrady, 1988a).

Several bills directing various government agencies to further study the feasibility of using enhanced degradable plastics, as well as other means of reducing plastic waste, were introduced in the Senate (e.g., S 2596 - Senator J.H. Chafee, October 25, 1986;

S2611 - Senator T. Stevens). Similar bills were introduced by the members of the House (H.R. 5380 - Mr. Hughes August 11, 1986; H.R. 5422 - Mr. Panetta, August 13, 1986, etc.). As a result of the legislative discussion, the Marine Plastic Pollution Research and Control Act of 1987 we signed by the President on December 29, 1987 (Public Law 100-220).

A related development is the U.S. ratification of the MARPOL Annex V, which will bring the convention into force by 1988. Final regulations implementing the Annex V, written by the U.S. Coast Guard, are expected to become effective by December 31, 1988.

These national and international developments have a direct short-term bearing on Naval activities. In spite of the exclusion of military vessels from MARPOL Annex V, it is advantageous for the Navy to be in a position to be capable of voluntarily complying with the provisions, if necessary.

1.4 Scope of Report

The research summarized in this report was carried out under Contract N61533-878-C-0002 (January to December 1988). Two major tasks were carried out: (i) a review of enhanced degradable plastic technology and its application to specific product areas, and (ii) tasks relating to development and assessment of environmentally disposable plastics for shipboard use. The first task, funded via the Navy under a cooperative agreement by the National Oceanic and Atmospheric Organization, will not be discussed in this report. The relevant review and conclusions, carried out in support of the Interagency Task Force on Marine Debris, were incorporated into the report published by the Task Force in May, 1987.

The second major task encompassed several activities that might conveniently be subcategorized as follows:

- Development of definitions, test protocols, and standards for the assessment of enhanced biodegradable plastics,
- Development and/or assessment of a range of potentially enhanced biodegradable plastics and demonstration of their performance under marine benthic conditions, and
- Study of the feasibility of developing materials that may be used in the short-term by Navy, in conformity with MARPOL Annex V provisions.

The scope of the project as described in these tasks addresses an area where very little scientific information is available, namely the development and assessment of biodegradable plastic technologies. While the concept of rapidly degradable plastics is not new, only a few of the technologies have been developed to the point of commercialization.

None of the commercial sources has data on the performance of their product under marine conditions. Thus, the existing technologies need to be evaluated for performance at sea and, where necessary, innovative new technologies developed to ensure adequate performance. The studies therefore range from field (marine) evaluation, accelerated laboratory evaluation, and support of standards development, to studies on promising approaches to enhancement of biodegradability of blends. The program of research is directed at narrowing the obvious gap in knowledge in this research area.

Addressing these questions is without doubt important to the Navy. Naval vessels rely on a variety of plastic materials that eventually enter into the ships' waste streams.

In its long-range strategy on waste management, the Navy will rely on a broad spectrum of technologies. Enhanced biodegradable plastic is likely to be a useful addition to these. It is convenient to be able to use an enhanced biodegradable plastic film material (with its multitude of desirable properties of economy, inertness, strength, and clarity) even under conditions where the discharge of regular plastics into the ocean is restricted.

2.0 Development of Definitions

A major activity within the current research program is the assessment of biodegradability under marine exposure. To be able to meaningfully compare the test results on the performance of different types of plastics (or results on the same plastic material generated by different experimenters), it is important to have Standard Test Protocols. Furthermore, the question of the degree of enhancement of environmental degradation desirable in plastic films and other products has to be eventually addressed. How much enhancement of the degradation is sufficient for a plastic material to be deemed environmentally acceptable? Such definitions and standards are not currently available. The need for the development of such definitions, standards, and test methods, before the enhanced degradable technologies can enjoy widespread use, was pointed out by a recent GAO report [GAO 1988].

A prerequisite for standards development is the evolution of explicit definitions to describe the phenomena. As a potential user of this technology, it is advantageous to the Navy to encourage these developments and to actively participate in the ensuing discussions to ensure that the requirements of the Navy are not compromised in the process.

The American Society forTesting and Materials [ASTM] is the foremost standards-making organization in the world and is particularly suited to address this question. An attempt was made to convince the organization as to the need of addressing the issue without delay. This was achieved by the Principal Investigator addressing ASTM's Committee D20.50 during their winter meeting in Boston in March 1988. Following the presentation and discussion, a study group was created to research the issue.

The Principal Investigator followed up the inital activities during the ASTM meeting in Toronto in October 1988 by participating (along with the Navy Project Manager) in a discussion on definitions. ASTM is currently pursuing the issue as an official activity of the subcommittee with three task groups studying definitions, photodegradable test methods, and biodegradable test methods. A writeup introducing the technology to the members was submitted to the official ASTM journal and was recently published [Andrady, 1988b].

In response to the discussion on definitions, an attempt was made to define several keywords commonly used in describing the materials in question. These definitions have been submitted to the ASTM task groups for their consideration and are expected to form a good starting point for the evolution of the final versions. The definitions are:

- Deterioration: The physical breakdown of a material into fragments.
- Degradation: The change of the chemical nature of a material as a result of chemical reactions, resulting in the loss of strength and/or other useful physical properties of the plastic material.

NOTE: In the case of plastics, the degradation process often results in the deterioration of the material. However, this need not necessarily be the case. Early stages of environmental degradation may not necessarily accompany deterioration.

- Photodegradation: Degradation that is brought about by the action of light (particularly in the ultraviolet region of the sunlight spectrum)
- Biodegradation: Degradation that is brought about by the action of living organisms using the material as a source of nutrients.

NOTE: This definition actually requires the microbes or other organisms to enzymatically degrade the plastic.

- Biodeterioration: Physical deterioration of the plastic by living organisms.
 NOTE: This process is a physical process where size reduction of the plastic may take place without actual biodegradation. Physical fragmentation may take place for instance, due to plant life growing on plastic surfaces, and animal life (particularly insect borers, etc.) attacking the material.
- Environmental Degradation: The degradation of a material in the environment due to the action of light, microbes, thermo-oxidation, hydrolysis, etc.

NOTE: Environmental deterioration invariably follows the environmental degradation of plastics.

NOTE: In nature, degradation processes are not necessarily effected by a single mechanism. Often two or more mechanisms act concurrently. Thus, for instance, an "enhanced photodegradable plastic" is one where the degradation is brought about predominantly (but not necessarily exclusively) by light-induced reactions.

It is particularly important to distinguish between biodegradation and biodeterioration, the latter term being proposed to refer exclusively to the fragmentation process. In the case of starch-based technologies (to be discussed later in the report), the change is one of biodeterioration of the material brought about by the microbial degradation of the starch fraction. It is not a true biodegradation because the polymer fraction is not enzymatically degraded to an appreciable extent but merely weakened and fragmented. In contrast, speciality polymers such as poly(caprolactone) are truly biodegradable.

3.0 Experimental Methodology

3.1 Sample Preparation

3.1.1 Materials

The low-density polyethylene used was USI NA 212, a resin that can be extrusion blown at relatively low melt temperatures (to minimize the possible thermal degradation of starch). The ethylene - vinyl acetate copolymer used was USI UE 633, which was 17 to 20 percent vinyl acetate.

Several different types of organic additives were used in the middle layer of the A-B-A coextruded, three-layered film samples. The origin and a brief description of each sample type are given below.

CORN STARCH: Corn starch is the additive of choice because it is inexpensive and readily available. Corn is the predominant type of starch produced in the United States and is recovered from the seed by a wet milling process. Starch is a mixture of at least two main types of biopolymers -- amylose and amylopectin. Amylose is a polymeric carbohydrate with anhydroglucose repeat units joined together by α (1-4) glucosidic bonds. Linear chains may have a degree of polymerization of 200 to 2,000. Amylopectin molecules are similar in structure to the amylose, except that a few of the repeat units branch off the C-6 position via α (1-6) bonds, with each branch consisting of 20 to 30 repeat units. Regular corn starch contains up to about 20 to 28 percent amylose, while varieties of corn also exist that yield much higher levels (up to 70 percent) of amylose. An average granule of corn starch is smaller than 100 μ m in diameter, and the material can absorb up to nearly 40 percent by weight of water vapor.

STARCH-CONTAINING MASTERBATCH: This masterbatch is commercially available in the United States and Canada, and the variety used in this study is manufactured from regular corn starch that is treated with a silane to render it hydrophobic. It is then dried until the moisture content is < 1 percent, and an unsaturated fatty acid additive is compounded into it. The masterbatch used was based on linear low-density polyethylene and contained about 43 percent of starch by weight.

CELLULOSE: Cellulose flock (powder) derived from cotton was obtained from a commercial source and was a white free-flowing powder that was dried prior to use.

Cellulose is the most widely occurring natural biopolymer. It is a polymer of ß D-glucopyranose units, with a degree of polymerization of several thousand. Unmodified cellulose derived from cotton has a tensile strength of about 200 to 800 MPa and an extensibility of 12 to 16 percent.

CORN COB: Powdered corn cob was obtained from a commercial supplier. The material derived from the woody portion of the cob is rich in polysaccharides (47 percent

cellulose, 37 percent hemicellulose, and 37 percent pentosan). The material absorbs up to 133 percent water and has a bulk density of 20 to 30 lbs/cubic feet.

3.1.2 Processing

Compounding: The additives were compounded into the respective resins to obtain a masterbatch, using a 40-mm twin screw compounding extruder with a screw L/D of 20:1 with 10 HP drive. The concentration of additive was adjusted to obtain a masterbatch which contained 20 to 50 percent of the additive. The extrudate was water cooled, pelletized, dried, and extruded immediately after compounding..

Extrusion: The extrusion of three-layer film samples was carried out in an assembly of two or three, 1 inch extruders. A screw with L/D ratio of 24:1 and a 3-HP drive were generally used. The screw design used was Flgts.Feed = 8, Trans. = 8 and meter. = 8. A breaker plate was used with no screens. A single extruder was used in the extrusion of single-layer films.

A bottom-fed coextrusion film die was used with a blown film tower, blower, and a torque winder. The system was well purged using virgin resin before each compound was run. All masterbatches were dried in a resin drying oven at 80 °C for several hours immediately prior to extrusion. The blown film samples were stored at ambient temperatures in a dark room until testing. Table 3.1 gives the extrusion parameters employed.

In removing samples for testing, the film samples were cut off after discarding the initial segment of film on the spool that might have been exposed to ambient light during storage.

Two series of sample films were prepared in the course of the study: (i) series of three-layer coextruded samples with starch (and other degradable additives) in the middle layer, and (ii) series of polyethylene-polycaprolactone blend films. These were designated Series A and Series B, respectively. (A third, Series C, was comprised of film samples acquired from outside sources and was also exposed.)

Table 3.1. A Summary of Extrusion Blowing Parameters Used.

					Tempe	erature,	۰F			
Material	Layer	RPM		Barrel zo	one		Die	Melt	P (psi)	Amp
			1	2	3	4	Die	MEIL	(þsi)	
LDPE resin	A	20	275	300	300	300	280	299	400	2.5
starch + LDPE	B	30	275	280	280	280	280	264	800	5.0
LDPE resin	A	20	275	300	300	300	280	299	400	2.5
cellulose + LDPE	B	30	280	280	280	280	280	264	800	3.0
LDPE resin	A	20	275	300	300	300	280	299	400	2.5
corncob + EVA	B	30	280	280	280	280	280	267	900	4.0
EVA resin	A	20	280	280	280	280	280	289	400	4.0
starch + EVA resin	B	3 0	280	280	280	280	280	262	700	4.5
EVA resin	A	20	280	280	280	280	280	289	400	4.0
Cellulose +EVA resin	B	30	280	280	280	280	280	264	800	3.5
EVA resin corncob+EVA resin	A	20	280	280	280	280	280	289	400	4.0
	B	30	290	290	290	290	290	265	900	4.0

RPM - Revolutions per minute P - Pressure

3.2 Exposure of Samples to Marine Environment

The exposures were carried out at a coastal location on Biscayne Bay in Miami, Florida. Sample sets tied to securing ropes were placed on the bottom sediment under 8 to 10 feet of water. Figure 3.1 is a photograph of the exposure location, while Figure 3.2 shows the recovery of the test frame assembly for periodic sampling.

The samples were mounted on a GRP plastic frame that allowed an 8 x 5 inch area of the film to be exposed. Use of a frame ensured that the film samples were placed flat on the bottom sediment and were held wrinkle-free during the exposure. A 1-inch margin around the exposed film was covered by the two halves of the frame held together by brass bolts. Figure 3.3 shows a typical frame with the film sample mounted. The frames were



Figure 3.1. Site of Marine Exposure.



Figure 3.2. Frame Assembly with Film Samples.

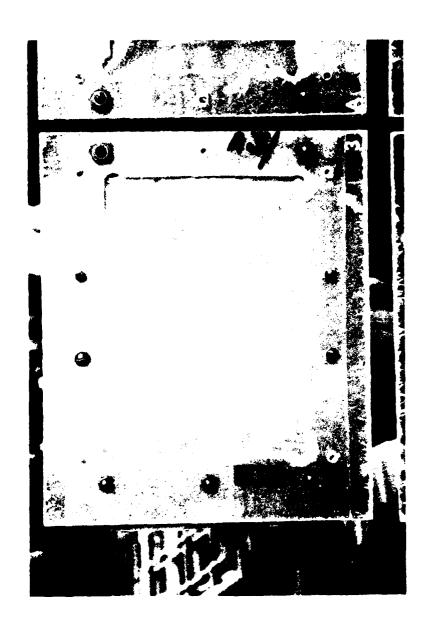


Figure 3.3. Exposure Frame with Film Samples.

affixed at one edge onto a rigid plastic strip for ease of handling during exposure. Sampling was carried out every 2 to 3 months for a period of 10 to 12 months. Exposed samples were removed from the water, taken off the frame, and allowed to dry at ambient temperatures. The dried films were returned to Research Triangle Institute (RTI) laboratories for testing.

3.3 Determination of Tensile Properties

Upon receipt of the exposed samples at RTI, tensile properties were measured on an Instron Model 1122 generally in accordance with ASTM D882, Tensile Properties of Thin Plastic Sheeting.

Specimens were cut with a Testing Machines, Inc. twin-blade cutter that gave consistent sample widths of one-half inch. Three to five thickness measurements of each specimen were read with a micrometer and the average reported. Sample thicknesses were measured to the nearest 1/1,000 mm. Due to the hydrophilic nature of the additives to the three-layer films (in the A series), the cut tensile specimens of these samples were dried overnight at 50 °C prior to Instron testing to remove trace amounts of water.

For Instron testing, air-powered grips with smooth 1 x 1.5 inch faces pressurized at 50 psi were used to hold the test specimens. A gauge length of 5.0 cm and a crosshead speed of 500 mm/min were employed. Five tensile specimens of each film sample were tested. The tensile strength was calculated by dividing the load to break by the cross-sectional area of the specimen (width x thickness). The percentage ultimate elongation was obtained by dividing the extension of the specimen at the break by the initial gauge length and multiplying by 100.

3.4 Gel Permeation Chromatography

Gel permeation chromatography (GPC) was used to determine the degradation of the polycaprolactone (PCL) component in the B series of films. Samples of the N-B8 (20 percent PCL) film exposed for 6 and 9 months were placed in chloroform at 50 °C to dissolve the residual PCL. The resulting solutions were approximately 0.15 g/100 mL PCL in chloroform. The solutions were filtered, and 200-μL injections were made into a Waters system chromatograph consisting of a Model M6000-A pump, a series of five columns, and a Model R401 differential refractometer detector. The columns were packed with Ultrastyragel with pore sizes ranging from 10⁶ to 10² A. The pore size decreased by a factor of 10 in successive columns.

4.0 Experimental Studies on Assessment of Enhanced Degradability

The present experimental effort was aimed at (a) assessing the degradability of plastic films based on existing semicommercial rapidly degradable technologies, under marine exposure conditions; (b) developing the more promising approaches to improve performance in biodegradable film applications; and (c) design of accelerated laboratory test methodologies to determine biodegradability.

4.1 Studies on Plastic-Starch Systems

A popular approach to rendering thermoplastics enhanced-deteriorable is to incorporate starch into the plastic matrix (Griffin, 1975; Otey et al., 1976, 1979, and 1987). Starch, a rapidly and completely biodegradable material, is expected to undergo preferential biodegradation on exposure to the environment (any biodegradation of the synthetic polymer within the same timescale will be so small as to be negligible). This process yields a porous plastic matrix with voids representing the volume fraction previously occupied by starch. Ensuing reduction in the mechanical integrity of the matrix leads to fragmentation or embrittlement, representing primarily a biodeterioration of the plastic. As the material is fragmented, the surface area exposed to the environment will increase, thereby favoring faster biodegradation of the plastic material.

The effectiveness of the approach will depend on the levels of starch in the composition, particle size of the starch, and the compounding-processing conditions. A high level of starch results in a higher volume fraction of voids being generated on exposure of the film to soil microbes and therefore leads to a faster rate of deterioration. However, the higher loading of hydrophilic starch in a hydrophobic thermoplastic matrix results in a marked reduction of the desirable mechanical properties of the plastic (see Table 4.1). The tensile properties are particularly affected, presumably as a result of the poor adhesion of the starch at the starch-polymer interface. This problem might be alleviated to some extent by pretreatment of the starch to render its surface hydrophobic. A silicone material or other proprietary coatings are used in commercial systems. In any event, the tensile properties of a plastic film are significantly reduced at levels of even 10 percent starch additive.

TABLE 4.1. Physical Properties of LLDPE Films (2.1 to 2.2 mil) with Various Amounts of Starch Additive.

Starch content [%]	0	3	6	9
Tensile strength [kg/sq.cm]	299	257	260	223
Ultimate elongation [%]	807	717	734	667
Dart impact [g]	192	160	174	118
Tear strength [g]	579	634	637	797

Source: Maddever and Chapman, 1987. Only the data relating to tests in the machine direction of extruded sheets are shown.

An alternative approach is to gelatinize the starch component and blend it into a thermoplastic mix, where at least one component is a copolymer with pendent carboxylic acid groups (Otey et al., 1976, 1979, and 1982). The starch component may either associate or even react with the carboxylic acid groups to yield a close mix between the starch and the thermoplastic. High levels of starch incorporated into blends of polyethylene/(ethylene-acrylic acid) copolymer using this technique also lead to reduced tensile properties. In unplasticized starch-thermoplastic films, the ultimate tensile elongation decreased by 71 percent on doubling the starch content from 20 to 40 percent (Swanson et al., 1988).

4.1.1 <u>Deterioration of Starch-Containing Single-Layer Plastic Films Under Marine</u> <u>Exposure Conditions</u>

Four types of commercially available films were used in the study. The films containing either 6.5 or 15 approximate weight percent of added starch were used as supplied by the manufacturer. C-4 is a polyethylene film containing approximately 15 percent starch in a low-density polyethylene [LDPE] matrix, while the C-5 film has the same starch level in an (ethylene-vinyl acetate) copolymer matrix. C-7, a relatively thicker film from a different manufacturer, contains only 6.5 percent starch in an LDPE matrix with an added thermo-oxidative degradation catalyst.

The thicknesses of these films are less than that of a film that might be used in the manufacture of heavy-duty plastic bags. Plastic bags for shipboard use and marine waste disposal applications would fall under the category of heavy-duty bags. The heavy-duty category of bags starts at the 20 lb (9.1 kg) capacity (FPA, 1971). These bags have a

minimum gauge length of 3 to 4 mils (0.07 to 0.10 mm) in LDPE (lower gauge thicknesses are achievable with linear LDPE). The upper range of heavy-duty bags includes those designed for a maximum load of about 40 lbs (of dimensions about 2.5 ft x 1.5 ft) and would generally be fabricated from polyethylene film at least 5 to 6 mils (0.13 to 0.18 mm) in thickness. In the case of starch-containing films with lower tensile strengths and/or elongations than unfilled polyethylene, an even thicker gauge of film might be necessary for fabrication of an equivalent bag. In this early study, thinner gauges of films were used, as most samples were provided by the manufacturers and as it was desirable to determine the general degradability of materials in the short term.

The extent of deterioration of plastic films can be monitored in principal using a variety of different test methods. With films intended for use in bag applications where tensile deformation is the prime mechanical characteristic of interest, ultimate tensile properties are best suited for the purpose. The tensile strength at the break (ultimate extension) of a composite (starch-filled) film, where one component is partially biodegraded, is a quantity that is difficult to interpret. Both the contribution of the starch fraction and the effect of void fractions created on partial degradation of starch need to be taken into account in interpreting ultimate strength data. As the starch is progressively removed, the strength of the film often increases. The ultimate extension is not dependent on thickness and is a more reliable measure of partial degradation of plastics. Table 4.2 summarizes the tensile results for the starch-polymer single-layer films. Detailed tensile test data for these samples are given in Appendix A (C Series Tensile Data).

The two samples containing approximately 15 percent starch lost about 66 to 69 percent of their ultimate elongation over a 6 month period of exposure. A corresponding loss for the C-7 film is difficult to quantify due to the relatively high standard deviation associated with the average ultimate elongation. However, it is clear that the rate of deterioration is much slower, due to the lower starch content (6.5 percent) of the film. The B-10 film is a commercial material submitted for evaluation and is probably also a starch-containing polyethylene. Being the thinnest film, it shows considerable deterioration in a 9-month period.

Based on the manufacturer's claims, a material containing 67 percent starch should completely deteriorate within a timescale of 6 months to 2 to 3 years when exposed to a soil environment. The C-7 material in particular is claimed to embrittle in the shorter period of time. On the basis of such incomplete data, the deterioration process is observed to be somewhat slower at sea than that claimed for soil environments. A more complete interpretation must await the data relating to longer exposure times (exposures are currently in progress). Direct comparison of the data in Table 4.2 is difficult due to the dif-

ferences in thicknesses of the samples. Figure 4.1 attempts a comparison of the data given in Table 4.2 by considering the percent ultimate extension retained at the end of 6 months of exposure for films of different thicknesses. (The exposure duration of 6 months was selected for the comparison on the basis of available data. Behavior at longer times might be different from that depicted in the figure.) However, the usefulness of a film depends both on the rapid deteriorability and good initial mechanical strength of the film.

TABLE 4.2. Biodeterioration of Starch-Polymer Single-Layer Films Under Marine Exposure Conditions.

Sample	T (mo.)	• • • • • • • • • • • • • • • • • • •		Ultimate extension (%)
B -1 (Pure LDPE)	0 2 6	0.072	167.09 [16.83] 147.51 [16.46] 142.81 [12.33]	241 [66] 220 [38] 167 [64]
C-4 (15% starch/PE)	0 3.5 6.0	0.055	67.60 [4.03] 84.29 [4.40] 102.31 [7.26]	373 [15] 93 [15] 114 [27]
C -5 (15% starch/EVA)	0 3.5 6	0.048	57.09 [7.62] 48.63 [7.13] 101.43 [8.76]	511 [48] 519 [26] 172 [14]
C -7 (6.5% starch/PE)	0 3.5 6	0.090	138.28 [3.95] 87.17 [8.40] 88.18 [9.29]	563 [14] 407 [63] 356 [200]
B-10	0 2 6 9	0.029	157.10 [19.76] 183.62 [18.85] 113.51 [11.04] 199.71 [20.60]	386 [34] 407 [26] 88 [28] 27 [18]

NOTE: The experiment is ongoing, and more data on longer exposure times are expected. C-4 and C-7 were LDPE films while C-5 was an ethylene - vinyl acetate copolymer. Sample B-10 was a material claimed to be "biodegradable," submitted to DTRC by a supplier of plastic films. [] indicates the standard deviation of the average values and is based on 3 to 5 measurements.

Film thickness is an important variable in determining the rate of biodegradation of the film. As the biodegradation of starch initiates at the surface on exposed granules of starch and works its way in, the rate of deterioration will become progressively slower for thicker films, at a given level of added starch.

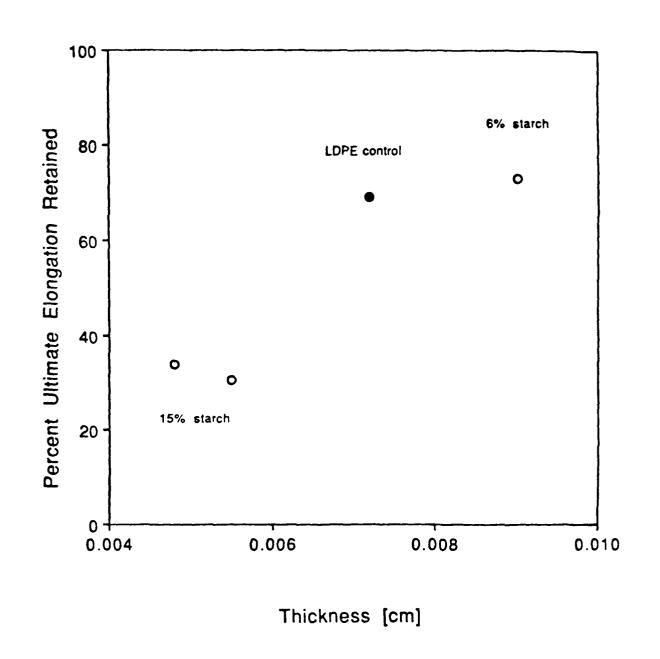


Figure 4.1. Percent Ultimate Elongation Retained After Six Months of Exposure for Single Layer Films.

Lower microbial activity in seawater (and bottom sediment) compared to that in soil or compost may lead to relatively slower biodeterioration of the material under marine exposure conditions. Slower utilization of the starch by the available microorganisms may also lead to a slower deterioration. However, the rates observed for coastal exposure might be different from those observed under deep-sea conditions. Lower temperatures, higher pH values, and the predominantly hyperbaric microbial populations in the benthic sediment might be expected to affect the rate of degradation. Experiments aimed at studying the biodegradation under deep-sea exposure conditions, for selected materials that biodeteriorate rapidly under present exposure conditions, are planned for a later phase in the study.

The initial tensile properties of polyolefin films containing relatively low levels of starch are high enough for general packaging and even grocery bag applications. In the case of heavy-duty bags, however, thicker gauges of film might be needed, and the resulting retardation of biodegradation of the starch component, particularly at sea, is likely to present a problem. Furthermore, the rates of deterioration of even the relatively thin films under marine exposure (see Table 4.2) are only moderate. Increasing the starch content to considerably improve the biodeteriorability while retaining the desirable tensile properties might prove to be difficult.

The surface quality and the "feel" of starch-containing plastics are also concerns. With increasing starch levels, the fractional area of the film surface covered by partially embedded starch granules increases. The resulting increase in hydrophilicity of the surface may affect the material's shelf life under humid storage conditions.

These difficulties might be overcome using two different approaches. One is to consider a flexible multilayer film where one or more discrete layers are made of a thermoplastic material (starch blend) while others are of the regular plastic. The other is to use blendable synthetic biodegradable polymers in place of starch. Preliminary experiments relating to these approaches are summarized in the following sections.

4.1.2 <u>Deterioration of Multilayer Plastic Films Containing Starch (and Other Degradable Materials) Under Marine Exposure Conditions</u>

The study was limited to films made up of three layers (A-B-A) with the layer containing starch or another biodegradable material sandwiched between two thinner outside layers. The outer layers of unfilled thermoplastic material were expected to (a) provide added integrity to the material, (b) improve its shelf life by eliminating the exposure of starch granules prone to biodegradation under ambient conditions, and (c) improve the surface characteristics of the film. With structural integrity being mainly provided by the

outer layers, the middle layer might be compounded with a relatively high loading of starch.

The coextrusion of the films described is described in Section 3.1. Table 4.3 summarizes the different varieties of the multilayer films prepared in the course of the study. The thickness of the outer layers was limited from one third to one half of that of the middle layer carrying the biodegradable additive. For successful biodeterioration, this outer layer should be as thin as possible. To maintain the necessary mechanical integrity, however, a certain minimum thickness of the outer layer is desirable. Selected ratios as shown in Table 4.3 were selected as the best compromise, at least for the purpose of this first study.

In water, the laminated structure might be disrupted due to several reasons: (i) The unequal imbibition of water by the two different types of layers, A and B, may lead to some local failure or delamination; (ii) the abrasion of the thin outer layers might expose the middle layer; or (iii) activity of marine macro-organisms on the outer layers might damage them sufficiently to allow the microbes access to organic material. In any event, marine microbes will gain access to the starch-laden (or other organic additive) middle layer, causing rapid biodeterioration. As the middle layer weakens, the multilayer laminate will progressively deteriorate in spite of the mechanical integrity afforded by the thin outer layers.

Four types of organic biodegradable additives were used in the preparation of these films:

- Corn starch.
- Commercially available starch-containing masterbatch (in LLDPE),
- · Cellulose flock, and
- Corncob powder.

The tensile test results for the different types of multilayer films are given in Tables 4.4 and 4.5 and are depicted in Figure 4.2. The tensile properties of films exposed at sea for different durations are given in Appendix B. Exposing a multilayer film to an aquatic environment may result in physical changes that lead to reduction in the ultimate elongation. The outer and inner layers of these films have widely different capacities for swelling by sorption of water. The internal stresses resulting from unequal swelling may even lead to local delamination and a change in the tensile properties. The as-extruded films do not, therefore, provide a good zero-time measurement. It is more meaningful in the case of these samples to regard the properties of the 2-month exposed sample as the starting point of the experiment. Tensile properties for the unexposed samples, however, are reported in Appendix C (A Series Unexposed Tensile Test Data).

Composition of Three-Layered (A-B-A) Films Containing Biodegradable Additives. Table 4.3.

Designation	Thickness (cm)	A Layer	B Layer	Thickness Ratio of ABA	Additive Content (~) (% by weight)
N-A1	0.0155	LDPE	EVA	1:3:1	_
N-A2	0.0151	EVA	EVA	1:3:1	
N-A3	0.0116	LDPE	Starch masterbatch*	1:1.5:1	18
N-A4	0.0159	LDPE	Starch masterbatch	1:3:1	26
N-A5	0.0133	LDPE	EVA:Starch masterbatch (1:1)	1:3:1	13
N-A6	0.0200	EVA	10% cornstarch in EVA	1:3:1	6
N-A7	0.0114	LDPE	10% cornstarch in EVA	1:1:1	3.5
N-A8	0.0230	EVA	20% cornstarch in EVA	1:3:1	12
N-A9	0.0207	LDPE	20% cornstarch in EVA	1:3:1	12
N-A10	0.0227	LDPE	20% cornstarch in EVA	1.5:3:1.5	10
N-A11	0.0223	EVA	20% cotton in EVA	1:3:1	12
N-A12	0.0209	LDPE	20% cotton in EVA	1:3:1	12
N-A13	0.0228	LDPE	20% corncob powder in EVA	1:3:1	12
N-A14	0.0320	EVA	20% corncob powder in EVA	1:3:1	12

^{*}Starch/linear low-density polyethylene masterbatch containing 43 percent starch.

LDPE =

Low-density polyethylene. (Ethylene/vinyl acetate) copolymer (17 to 20 percent vinyl acetate). EVA

Table 4.4. Multilayer Films - Tensile Data Summary.

		Tensile Strength (kg/cm ²) Ultimate Elonga				
Sample	Exposure Time (mo.)	Average	Standard Deviation	Average	Standard Deviation	
N-A1	2	125.8	8.7	863.0	75.9	
	4	80.4	4.6			
	6 8	83.0	5.7			
	8	114.0	5.2	671.0	19.4	
	10	108.5	9.1	653.3	59.5	
N-A2	2	172.4	4.3	1356.2	22.9	
	2 4	93.9	8.6	903.8	74.6	
	6	93.1	8.8			
	8	143.3	4.6	1147.6	85.4	
	10	71.7	3.6	714.3	70.4	
N-A3	2	66.2	7.4	409.3	44.8	
	4	57.9	7.5			
	6	69.7	5.2	361.1	50.5	
	8	66.5	4.4	350.0	121.7	
	10	73.6	6.1	357.2	55.2	
N-A4	2	59.7	5.5	510.1	117.3	
	4	53.9	5.1			
	6	60.0	2.5	360.0	126.7	
	8	57.6	3.3	338.3	64.7	
	10	63.5	4.1	304.4	113.1	
N-A5	2	97.1	4.1	807.1	97.7	
	4	75.8	6.5	530.5	37.6	
	6	87.0	8.7			
	8	90.6	5.1	697.1	67.7	
	10	69.9	5.2	492.2	22.0	
N-A6	2	58.8	3.3	8.008	28.7	
	4	50.7	3.9			
	2 4 6 8	59.6	3.8	773.3	131.3	
		62.6	2.0	789.3	57.4	
	1 0	66.8	1.6	829.9	57.0	
N-A7	2	64.8	4.2	175.2	16.9	
	2 4	58.0	4.1			
	6	61.7	4.5	130.4	16.1	
	8	62.6	5.1	153.8	6.3	
	10	61.8	1.8	128.1	13.7	

(continued)

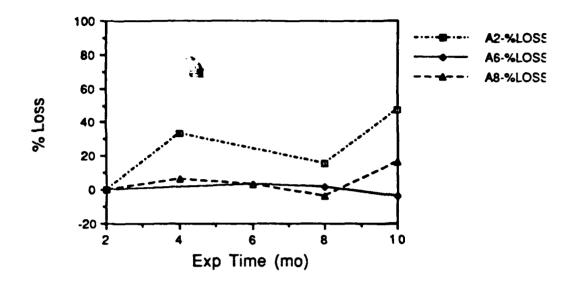
Table 4.4 (continued)

		Tensile Stre	ngth (kg/cm ²)	Ultimate E	longation (%)
Sample	Exposure Time (mo.)	Average	Standard Deviation	Average	Standard Deviation
AL A O	2	49.1	3.4	758.9	81.4
N-A8	2 4	47.4	3.4	710.3	103.3
	6	41.7	3.8	736.0	90.4
	8	48.6	3.7	785.4	63.1
	10	45.7	3.4	635.3	45.0
N-A9	2	50.1	3.7	443.3	42.8
	4	38.6	2.7	406.8	62.4
	6	46.2	0.9		
	8	50.4	2.9	386.7	35.4
	1 0	51.3	4.4	375.3	77.4
N-A10	2	58.3	3.5	437.3	52.8
	4	58.1	4.7		47.4
	6	61.0	1.9	508.8	47.1
	8 1 0	61.8 60.3	6.7 4 .5	456.9 433.7	87.0 35 .0
N-A11	2	62.2	5.6	938.3	66.9
(A-VII	4	56.5	5.1	749.1	112.3
	6	54.0	3.3	729.7	69.2
	8	55.2	5.7	872.9	91.5
	10	52.6	5.8	601.8	83.9
N-A12	2	56.7	5.1	436.6	21.1
	4	53.6	4.6	372.6	30.2
	6	57.8	3.5	436.3	32.3
	8	57.0	3.8	353.0	31.8
	10	62.2	2.7	401.7	46.4
N-A13	2	38.4	4.3	323.4	49.0
	4	40.1	2.4	460.7	48.6
	6	33 .1	3.6	328.6	10.0
	8 1 0	37.1 39.9	2.3 4.1	386.2	69.1
N-A14	2	36.8	6.6	867.8	7.2 62.4
	4 6	36.3 35.4	2.6 4.3	746.1	02.4
	8	35. 4 37.6	4.3 4.2	758.4	50.0
	10	34.0	2.2	593.2	79.9

Table 4.5. Multilayer Films - Ultimate Elongation Percent Decrease.

Sample	% Elongation		% Decrease*		
	Exposure Time (months)				
	2	4	6	8	10
N-A1	863.0	-	•	22.20	24.30
N-A2	1356.2	33.40	-	15.40	47.30
N-A3	409.3	•	11.80	14.50	12.70
N-A4	510.1	-	29.40	33.70	40.30
N-A5	807.1	34.30	•	13.60	39.00
N-A6	800.8	•	3.43	1.44	
N-A7	175.2	25.60	25.60	12.20	26.90
N-A8	758.9	6.40	3.02		16.30
N-A9	443.3	8.23	-	12.80	15.30
N-A10	437.3	•			0.82
N-A11	938.3	20.20	22.20	6.97	35.90
N-A12	436.6	14.70	0.07	19.10	7.99
N-A13	323.4			-	
N-A14	867.8	14.00	-	12.60	31.60

^{* %} decrease from 2-month value.



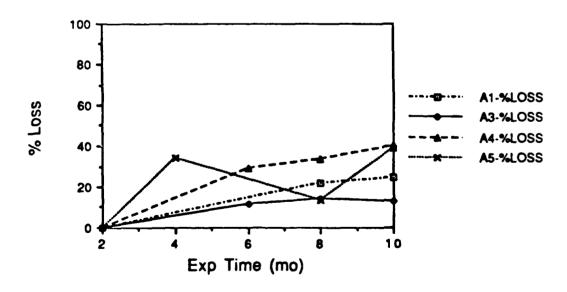
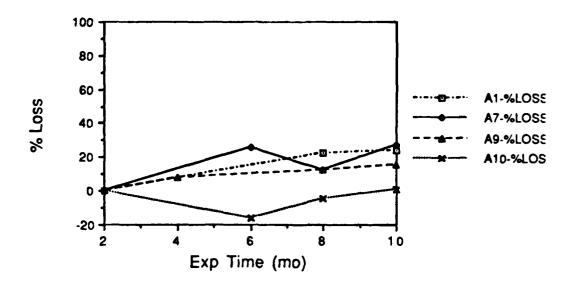


Figure 4.2. Multilayer Films - Percent Decrease in Ultimate Elongation.



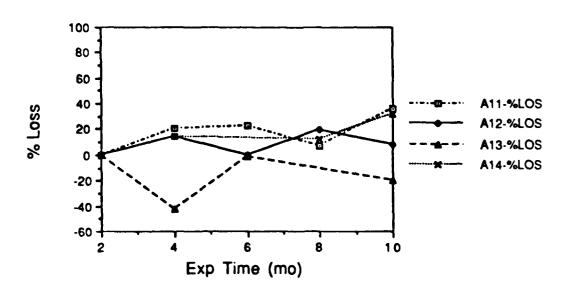


Figure 4.2 (continued)

The variability (scatter) in the tensile properties of plastic films exposed for several months in the marine environment is much higher than that for the unexposed film material. This might mainly be attributed to the activity of surface foulants. Samples exposed even for a period of several months showed extensive biofouling (see Figure 4.3). Unlike in the case of plastics floating in seawater, where algal fouling is predominant, the submerged surfaces also attract macrofoulants such as barnacles. In addition, unidentified fungal and other microbial colonies often covered wide areas of the film. The area of the film directly below a large patch of foulant would be subjected to a different microbial environment compared to a relatively unfouled portion of the same film. The resulting inhomogeneity may lead to uneven weathering of some additive-containing films. However, as the degree and nature of fouling depends on the nature of the film, level of additive, and the period of exposure, it is difficult to take this factor into account (by, for instance, sampling from only the unfouled regions of film). The test strips cut from the film in some cases did traverse such "weak" spots and yielded unrealistically low values for ultimate elongation. Data points based on such values are not shown in the figures.

Differences in the origins and grade of the base resins used in current A-B-A films and those discussed in the previous section preclude direct comparison of the data. In any case, only several samples are comparable even in terms of starch content. A-8 and A-10 are three-layer films with outer layers of (ethylene/vinyl acetate) copolymer (EVA) and low density polyethylene (LDPE), respectively. Both contain about 12 percent unmodified corn starch and might therefore be compared to the single-layer films C-4 and C-5. These three-layer films show about the same tensile strengths and somewhat higher ultimate extensions compared to single-layer films. However, being much thicker, they deteriorated much more slowly than did the single-layer films.

The coextruded films did have good tensile properties even at high loadings of starch in the middle layer. While some enhancement of deterioration was observed with specific samples, the three-layer films studied here had, in general, lifetimes longer than 1 year. Apparently, the thickness of the outer (A and C) layers needs to be reduced to effect deterioration within a shorter timescale.



Figure 4.3. Biofouling of Films Exposed under Marine Conditions.

4.2 <u>Blends of Commodity Plastics with Readily Biodegradable Synthetic Polymers</u>

The starch-containing plastic films discussed in the above section are based on the concept of blending a nonbiodegradable (or slowly degradable) plastic material with a readily degradable polymer, so as to render the composite material enhanced-biodeteriorable. While starch is inexpensive and readily degradable, it is also essentially incompatible with synthetic polymers. It is this incompatibility that is responsible for poor dispersion and lower mechanical strength of starch-containing films.

This general concept might be extended to a blend where both components are synthetic polymers but one of these is readily biodegradable in the environment. Several synthetic polymers are known to be readily biodegradable. While these speciality polymers are more expensive than the biopolymers such as starch, they afford better compatibility and may bring about the desired rates of deterioration at low levels of additives. It is therefore of interest to explore some of these materials to determine (a) their performance under marine conditions, and (b) their effectiveness and limitations.

For the purpose of the present study, a series of blends of polyethylene with polycaprolactone was studied. Polycaprolactone is commercially available (Union Carbide) in the United States and is blendable with polyolefins. The material is stable up to at least 225 °C, allowing it to be easily compounded into a thermoplastic system. Selected physical properties of the polycaprolactone used are given in Table 4.6.

TABLE 4.6. Selected Properties of Polycaprolactone P-700.

O || | |-CH2CH2CH2CH2CH2-C-O-]n

Melting point:

60 °C

Glass transition temperature:

-60 °C

Density (0 °C):

1.16 g/cc

Moisture content at 100% RH:

0.43%

4.2.1 <u>Deterioration of Polyethylene-Polycaprolactone Blends Under Marine Exposure</u> Conditions

Extrusion-blown films of polycaprolactone [PCL] in low-density polyethylene [USI NA 212 LDPE] were exposed to the marine sediment. The compositions covered the range

of 0 - 20 weight percent of polycaprolactone in the blend. Data collected during 0 to 9 months of exposure are available at this time and are shown in Tables 4.7 and 4.8 and Figure 4.4. Detailed tensile data are given in Appendix D. Tensile test results for unexposed polypropylene/polycaprolactone blend films were also obtained and are shown in Appendix E. This latter series of blends have not as yet been exposed or tested.

Films of polycaprolactone-polyethylene blends showed high initial tensile strengths and ultimate elongation. The values were generally higher than those obtained with polyethylene-starch blends. Increasing the weight fraction of PCL in the blend up to about 20 percent did not result in an appreciable increase in the tensile strength of the material, but the ultimate elongation increased significantly when the weight percent exceeded about 10 (see Table 4.7). Even at 2 to 6 percent levels of PCL in the blend, a significant increase in the rate of deterioration was observed. At 8 to 10 percent levels, the deterioration rates were markedly higher than those of control films, and the initial tensile properties were not too different from those of plain polyethylene films. At these PCL levels, the blends can be easily processed and used to fabricate heavy-duty plastic bags.

Films with a PCL weight fraction of higher than 20 percent showed high extensibility, in agreement with previous observations (Union Carbide). On exposure to the marine sediment, the material developed tears or cracks in the flow direction within 2 to 4 months. This might be a result of inadequate mixing at the higher levels of PCL in the blend, resulting in narrow PCL-rich streaks in the flow direction. This phenomenon may to some degree have contributed to the apparent rapid degradability at PCL levels > 10 percent.

Table 4.7 shows the variation in ultimate tensile properties with exposure time for these blends. Both the tensile strength and the ultimate elongation of the films decreased with the time of exposure. As indicated elsewhere, the latter quantity affords a better measure of the extent of deterioration. During the 9-month period of observation, the ultimate elongations decreased by 55 to 75 percent depending on the weight fraction of PCL in the blends. (Data for longer exposure times are expected shortly.)

The rate of biodeterioration of the blends might be compared to those of starch-filled single-layer films by superimposing the data in Table 4.8 onto Figure 4.1, which presents the percentage ultimate elongation retained after 6 months of exposure. Figure 4.5 shows the superimposed data; the blends compare favorably with the starch-based film technology. The 6-month data points are used for the comparison to be consistent with data relating to single-layer films.

Extensive fouling was observed on these films, as shown in Figure 4.3. Because a study of the nature of microbial populations was not attempted, the composition of the

Table 4.7. Polyethylene/Polycaprolactone Films - Tensile Data Summary.

Sample	% PCL		Tensile Stren	gth (kg/km ²)	Ultimate Elo	ngation (%)
		Exposure Time (mo)	Average	Standard Deviation	Average	Standard Deviation
N-B1	0	0	167	17	241.3	65.7
		2	148	16	219.6	37.5
		6 9	143	1 2 5	167.4	63.5
		9	106	5	108.0	11.8
N-B2	2	0	171	13	353.4	30.5
	_	2	150	7	281.8	49.5
		6	123	12	150.1	36.7
		9	112	11	135.7	15.9
N-B3	4	0	163	7	345.9	36.5
		2	125	9	140.5	29.6
		6	116	3	136.8	21.0
		9	106	11	75.0	7.7
N-B4	6	0	167	2 1	355.5	25.1
		2	163	10	303.5	21.8
		6	142	3	173.9	10.1
		9	117	8	80.2	15.5
N-B5	8	0	179	5	332.0	24.0
		2	145	12	202.6	46.5
		6	126	5	117.7	28.1
		9	120	13	82.8	22.2
N-B6	10	0	169	7	285.2	22.5
		2	147	8	190.8	39.4
		2 6 9	112	10	82.1	3.2
		9	115	16	72.5	20.4

(continued)

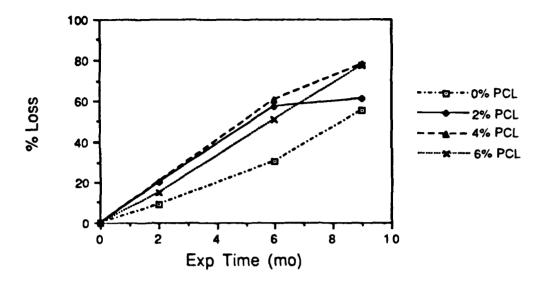
Table 4.7 (continued)

Sample	% PCL	•	Tensile Stren	ngth (kg/km²)	Ultimate Elo	ngation (%)
		Exposure Time (mo)	Average	Standard Deviation	Average	Standard Deviation
N-B7	15	0 2 6 9	191 160 125 146	1 0 7 3 1 3	360.9 195.2 90.2 91.2	33.4 22.3 12.3 19.4
N-B8	20	0 2 6 9	184 158 123 131	20 13 8 3	541.1 341.1 76.6 90.5	54.3 26.8 4.2 16.4

Conditions:
Gauge Length: 50 mm
Crosshead: 500 mm/min

Table 4.8. Polyethylene/Polycaprolactone Films - Ultimate Elongation Percent Decrease.

	% Elongation		% Decrease	•	
Sample		Exposur	e Time (months	5)	
	% PCL	0	2	6	9
N-B1 N-B2	0 2	241.3 353.4	8.99 20.3	30.6 57.5	55.2 61.6
N-B3	4	345.9	59.4	6° 5	78.3
N-B4 N-B5	6 8	355.5 332.0	14.6 39.0	51.1 64.5	77.4 75.1
N-B6 N-B7	1 0 1 5	285.2 360.9	33.1 45.9	71.2 75.0	74.6 74.7
N-B8	20	541.1	37.0	85.8	83.3



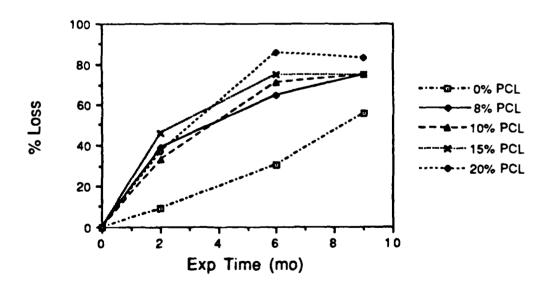


Figure 4.4. Polyethylene/Polycaprolactone Films - Percent Decrease in Ultimate Elongation.

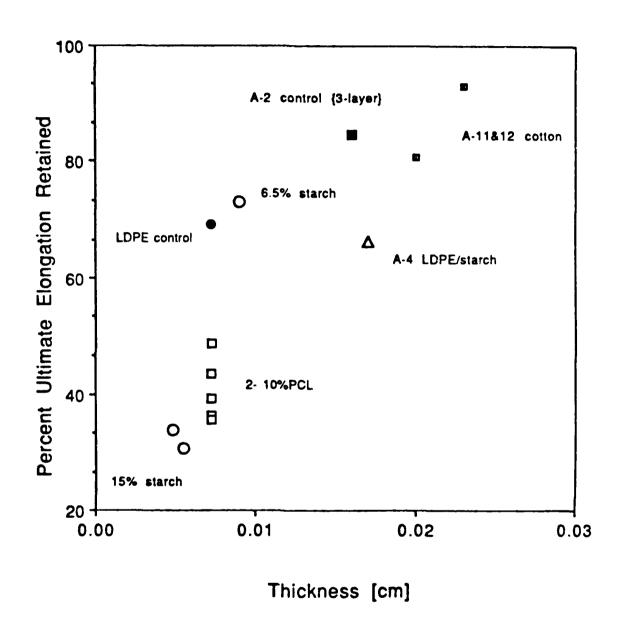


Figure 4.5. Comparison of Percent Ultimate Elongation Retained After Six Months of Exposure for Synthetic Polymer Blends and Starch-Filled Single Layer Films.

foulant layer is not known. Macrofoulants such as barnacles, however, were detected on the film surface.

4.2.2 Characterization of Polycaprolactone Molecular Weight Loss by Gel Permeation Chromatography.

To confirm that true biodegradation (as opposed to biodeterioration) of polycaprolactone did occur in the blends, molecular weights of the residual polymer was determined for two of the samples. Gel permeation chromatography was used to determine the molecular weight of the polycaprolactone in samples NB-8 exposed for 6 and 9 months. As illustrated in Figure 4.6, the data show a very significant decrease in the average molecular weights of the residual PCL fraction. Some of the polymer would have been more extensively degraded and lost as leachable oligomers. The determination therefore overestimate the average molecular weights for the degraded material.

4.3 Other Miscellaneous Polymers

Two other synthetic polymers were included in the exposure studies. These were films of copolymers of lactic acid (three types) and poly(propylene carbonate). The lactic acid copolymers were found to biodeteriorate rapidly under the exposure conditions. As seen in Table 4.9, the materials did not survive the first sample collection period at the end of the second month. The poly(alkylene carbonate) films used were supplied by the manufacturer as thin films (0.003 to 0.004 cm). These were too fragile for exposure under water. The data reported in the table should therefore be regarded as approximate and preliminary.

TABLE 4.9. Durations of Marine Exposure Carried Out During Reporting Period.

Designation	Material	Duration	Status
B-1, A-1, A-2	Unmodified plastic film	1 year	+
Series A	Biopolymer /plastic films	1 year	+
Series B	Polymer blend [LDPE/PCL]	> 9 months	+ *
C-1	Poly(propylene carbonate)	> 2 months	+ #
C-2, C-3, A-16	D,L lactide/glycolide copolymers	1 - 2 months	-

^{+ =} Samples intact.

Samples completely embrittled.

Ongoing experiment.

^{# =} Discontinued.

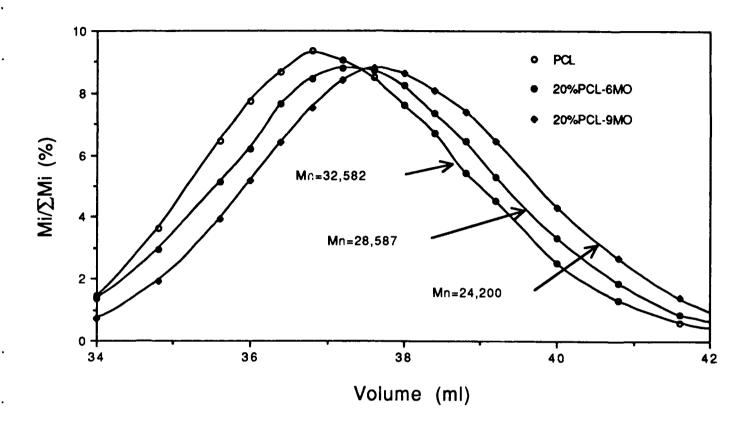


Figure 4.6. GPC Curves for Selected PE/PCL Films.

Lactide/glycolide copolymers were the most rapidly degradable materials exposed. The entire sample, with the exception of the section of the sample sandwiched between the sides of the frame, disappeared within 1 month (A-16) or 2 months (C-2 and C-3) of exposure. Presumably, the material embrittled, broke up into small fragments, and was washed away. The fragments under the frame were 2 to 3 cm wide and were brittle. This material is apparently a very rapidly biodegradable polymer. However, its very high cost at this time makes it an unrealistic candidate as a material for plastic bags.

The poly(propylene carbonate) films (C-1) tested were thin films supplied by the manufacturer. The films were too thin to withstand exposure and were found to be torn at the first sampling period. Exposure of the material was discontinued, and thicker samples under fabrication will be tested at a later date.

4.4 Design of Accelerated Biodegradation Equipment

While exposure to the environment under field conditions is obviously the best means of assessing the biodegradability of a given sample of plastic material, it is also an expensive and time-consuming technique. Furthermore, the rate of deterioration on outdoor exposures depend on the natural conditions prevailing during the experiment. Fluctuations and extremes in water quality, ambient temperature, and microbial growth cycles are hardly reproducible every time the exposure is carried out. While outdoor testing will eventually be needed to fully demonstrate the degradability of the more promising formulations, its use as a screening test for candidate technologies is wasteful and time-consuming.

A laboratory accelerated degradation procedure is an attractive alternative for easy and rapid assessment of degradability/deteriorability of candidate samples under well-defined exposure conditions. Testing under laboratory conditions allows the acceleration of the deterioration process by intensifying one or more of the factors that bring about the degradation; it also allows for faster evaluations. It is, however, crucial to ensure that acceleration is not carried to the extent that the physical and biochemical processes taking place under the accelerated conditions are not representative of the environmental degradation processes.

In the case of marine biodegradation, in the absence of light, the following factors might be expected to directly affect the rate of deterioration:

 The microbial population density: The availability of appropriate species of microbes in sufficient numbers in the vicinity of the test sample is a primary requirement for biodegradation. In the case of starch, a wide variety of microbes capable of using the substrate as a carbon source is to be expected in both marine and soil environments.

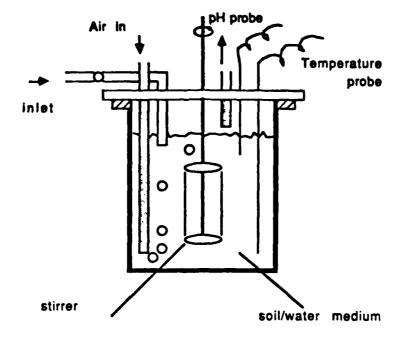
- Oxygen availability: The growth of the microbial population will depend upon the availability of air. In the case of aerobic species and facultative aerobic species, the presence of oxygen determines the microbial density in the medium.
- Temperature: Temperature also determines the rate of growth and assimilation of starch by microbes.
- Acidity of the medium: Seawater, for instance, is slightly alkaline and therefore supports species that may not grow in a slightly acidic soil environment. The pH of sea water is also dependent upon the depth to some extent.
- Availability of nutrients: The availability of trace nutrients and even the availability of alternative carbon sources can have an impact on the development of the microbial population and therefore on the rates of degradation.

The present apparatus (a modified fermenter) was set up taking the above basic factors into consideration. It consists of three glass biodegradation chambers placed in a thermostatted water bath. Each chamber has several inlets, outlets, and stirring facilities. Appropriate sensors for the measurement of temperature, pH, and airflow are fitted in each chamber. The signals from the sensors are recorded on disk via a dedicated interface system.

Samples are placed in soil, seawater, or enriched media in the chambers and allowed to deteriorate under suitable temperature and pH conditions. It is crucial to make sure that the the conditions remain constant throughout the exposure, which may last for several days or weeks. Samples that are withdrawn, washed, and dried at intervals will be tested using an appropriate mechanical property determination. Aerobic degradation of starch might also be monitored via the measurement of the carbon dioxide produced. In a future modification, the carbon dioxide level in air flowing through each chamber will be recorded using an infra-red absorbance detector, throughout the exposure period.

In accelerating the process, increased temperatures, good aeration, and an enriched culture of microbes will be used. The latter might be a mixed culture of soil microorganisms or a sewage sludge innoculum. The microbial activity of the medium will be estimated from the initial biological oxygen demand [BOD] of the material in the chamber. Monitoring the increase in carbon dioxide production over that of background level will give an indication of the extent of biodegradation (aerobic processes) that might be correlated to the decreases in the physical properties of the plastic films.

Figure 4.7 shows a schematic diagram of a single chamber, and Figure 4.8 shows a photograph of the equipment.



a) Biodegradation Chamber

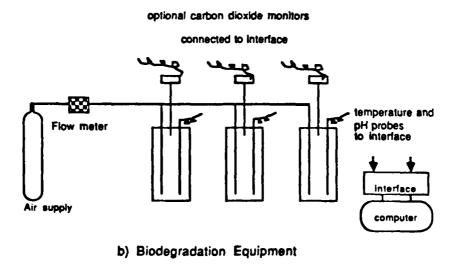


Figure 4.7. Schematic Diagram of Accelerated Biodegradation Equipment.

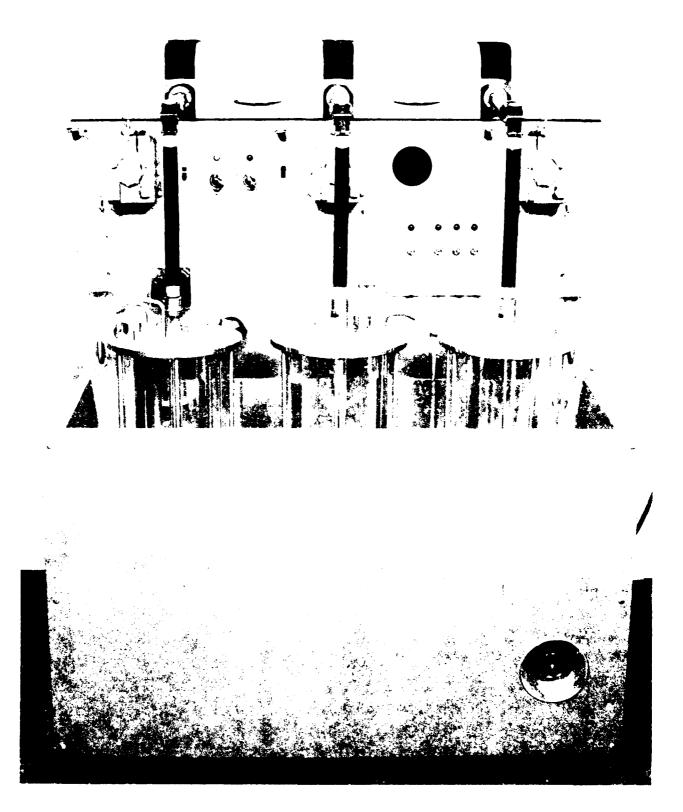


Figure 4.8. Accelerated Biodegradation Equipment.

5.0 Conclusions and Directions for Further Work

5.1 Conclusions

- Development of definitions, standards, and test protocols relating to enhanced degradable plastics is an urgent need. The Navy has initiated a discussion of the subject within the ASTM (American Society for Testing and Materials) and continues to contribute to the process from the point of view of a potential user of the technology.
- Single-layer plastic films with starch incorporated as a filler degraded at sea but at only moderate rates depending on the thickness of the film material. The plastic film materials tested were not completely embrittled in the 6 to 9 months of exposure. In view of the current ecological concerns, material with a lifetime of only a few weeks at sea is preferable for Navy use. While faster biodegradation might be achieved with thinner films, heavyduty plastic bags require the use of thicker gauge films to ensure good mechanical strength.
- Coextrusion of three-layer films, with the middle layer containing high levels of various biodegradable fillers and the outside layers consisting of thin unfilled plastic for increased mechanical integrity, was carried out. These films also biodeteriorated at rates equal to or slower than those of single-layer films. While some improvement in the rate might be obtained with thinner outer layers, the lifetimes (to embrittlement) is likely to remain longer than the period of observation of 1 year.
- Given the requirement for fast deterioration (in a time span of about 4 weeks in sea water), the study shows that the presently available starch-based systems tested, will not meet the Navy needs for a rapidly biodegradable plastic film. As such, research on these technologies should be de-emphasized in 1989.
- Films of various blends of polycaprolactone-polyethylene, containing up to 10 percent of the biodegradable polycaprolactone polymer, did biodeteriorate significantly during 6 months of exposure at sea. The blends produced films of good surface properties, good mechanical strength, and moderate biodeteriorability at sea. At levels exceeding 15 percent polycaprolactone in the blend, the films were weaker and developed weak spots. These blends are more promising candidates for enhanced biodeteriorable films and deserve further study. However, these polyesters will not allow biodegradation in a time scale as short as a few weeks.
- A laboratory-accelerated biodeterioration equipment has been put together for further work on the correlation of field data with data from laboratory-

accelerated degradation experiments. The equipment allows for close monitoring of temperature and pH during the full duration of exposure to a microbe-enriched environment. Capability to periodically monitor the carbon dioxide concentration in the air passing through the biodegradation chamber is currently being planned.

5.2 Directions for Future Work

- A reassessment of the maximum lifetimes for plastic films intended for shipboard (and other Naval) use is recommended, taking into account the Navy requirements, as well as the current legislative, technical, and environmental discussions on the issue of plastics in the environment.
- Studies are needed on the effect of marine exposure on untreated plastic films. Plastics are generally considered to be nonbiodegradable materials. Some of the available data, however, seem to suggest that plastic films lose their tensile properties to a measurable extent over a 6 to 10 month period of exposure in seawater. While this might be due to a physical mechanism such as surface damage to the film (form macrofoulants, etc.), which will affect its ultimate elongation, the phenomenon deserves further investigation.
- Use of chemically modified starches to attain faster degradability and better tensile properties should be investigated. A wide range of chemically modified starches are available. Those modifications that yield slightly more hydrophobic starches are likely to yield higher initial strengths when blended with thermoplastics. The ease of biodegradability of these blends, in particular, needs to be studied.
- Further studies on polycaprolactone-polyolefin blends should be undertaken, with an extension of the experiments to polyethylene and ethylene-vinyl acetate copolymers. A study of copolymers of polypropylene and polycaprolactone, which are available semi-commercially and might prove to be suitable for film extrusion and undergo rapid deterioration, is particularly needed.
- Studies should be done on water-soluble synthetic polymers to reduce their lifetimes at sea from months to several days (or even hours). Biodegradability of water-soluble polymers, particularly poly(ethylene oxide), polymeric cellulose derivatives, and poly(vinyl alcohol), could be a focus on such studies.
- Studies might also be done to see whether chitin-derived polymers could serve as ingestible, fully biodegradable and/or water soluble materials for use as packaging films. Such films will be dischargeable into the sea even under the provisions of MARPOL Annex V. If successful, this approach allows the Navy a short-term strategy for addressing the restrictions of Annex V.

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Appendix A C Series Tensile Data

1	Load at Visido Load at Broak		A at Break	Thickness*	Tensile	Strength at Yield	PIEX	Tensile	Strength at	9 5 6 5 6 5 6 5 6 5 6 5 6 5 6 5 6 5 6 5	3	% Elongation at Break	Best Best Best Best Best Best Best Best	1
N-C1-0-1		<u>.</u>		0 0033	, a	•			•			•		
N-C1-0-2			5.4	0 0038				371.1			10.			
N-C1-0-3	•	1 50	0,4	0 0047				253.0	279.3	64.2	0.0	6.3	1.7	
N.C1.0.4	•	1 .	9.8	0.0040				222.9			7.0			-
N-C1-0-5	•	+	9.7	0.0043				270.4			4.7			•
N-C1-2MO-1	•	1.40	•	0.0045				242.8			7.2			•
N.C1.2MO-2		1 90	4.2	0.0067				224.9			+ 0			
N-C1-2MO-3		100	9.6	0 0044				194.2	193.6	57.7	7.6	7.1	7.1	
N-C1-2MO-4	•	0.67	2.6	0.0047				112.4			5.2			•
N-C1-2MO-5				0.0042										
N.C4-0-1	0.45	0.50	193.3	0.0055	63.7			71.6			386.6			<u>د</u>
N.C4-0-2	44.0	97.0	173.0	0.0055	62.3						347.2			٠ •
N-C4-0-3	0.45	0.49	166.3	0.0055	4.40	62.9		69.4	97.6	0.4	376.6	373.4	15.4	0 .
N-C4-0-4	0.43	0.45	187.1	0.0055	9.19			7 79			374.2			O.
N.C4-0-5	0.45	0.50	191.2	0.0056	97.0			70.3			382.4			٠ <u>.</u>
N-C4-3 5MO-1		0.0	56.0	0.0058				13.7			113.8			•
N-C4-3,5MO-2	•	0.60	51.5	0 0056				7.7			103.0			•
N-C4-3,5MO-3		0.59	43.1	0.000				77.4	84.3	4.4	86 2	93.3	14.9	
N-C4-3 5MO-4	•	0.64	43.4	0.0058				80.8			9.99			•
N-C4-3.5MO-5	٠	0.65	36.3	0.0057				1.60			76.6			
			•								:			•
								4 001			124.4			
	•	0	72.3	0.0047				9 011	102.3	7.3	144 6	114.4	28.0	
N-C4-BMO-4	•	990	0.50	0.0050				103.3	}	!	130.0			•
N-C4-010-5	•	0.0	42.0	0.0052				8 06			0.7			•
N-C5-0-1	•	0.33	234.0	0.0048				53.3			468.0			•
K-C2-0-2	•	0.40	260.8	0.0048				92.0		i	521.6		,	-
K-0-50-3	•	90.0	200.5	0.0046				65.0	27.1	9.	0.00	510.7	C: /*	
4-0-60-E	•	0.32	2,40.0	200.0				4.0			0			• •
N-C3-6-3	•	5	0.00	• • • • • • • • • • • • • • • • • • • •				0.06						•
N-CS-3,5MO-1	•		274.0	0.0050				56.7			540.0			•
N-CS-3.5MO-2	٠	0.26	164.0	0.0051				38.4						
N-CS-3.5MO-3		0.29	184.0	0.0052				43.2	9.9	7.1		518.7	25.6	-
N-CS-3.5MO-4	•		250.1	0.0049				51.4			507.2			-
N-CS-3.5MO-5			254.0	0.0051				52.5			0.00			-
N.CS-6MO-1		0.67	110.6	0.0047				111.5						-
N.CS-6MO-2		0.50		0.0056										•
N-CS-BMO-3	•	0.57	79.2	0.0044				102.1	101.4	•	158.4	172.7	14.1	•
N-CS-8EO-4	•	5.0	93.3	0.0053				9 0			106.6			-
N.CS-BEO-S		0.63	•	0.0048				102.0			173.2			•
N-C7-0-1	1.12	1,40	275.0	0.0091	96.5			138 0			551.6			٥
N-C7-0-2	1.09	7.0	287.1	0600.0	95.4			140 0			574.2	•		٩
R-C7-0-3	1.0	1.57	201.4	0.0000	1.16	1 20	1.3	137.4	138.3	0.4	562 8	563.4	13.0	.
N-C7-0-4	1.1	1 53	274.1	0 0091	0.96			132.4			548.2			٥
N-C7-0-5	1.00	1,65	280.2	0.0091	93.4			142.8			580.4			م

Appendix A - Navy C Series Tensile Data

		407.3					356.2		
3558	428.4	358.4		486.6	136 6	556 8	559 0	344 8	
		•					6.9		
		87.2					86 2		
7.8.7	86 5	79.2	1 50	96.3	912	7 86	7 70	83.9	7 8 7
		9.					5.8		
		76.1					81.8		
75.0	79.2	75.0	74.6	9.97	85.5	77.9	62.5	79.9	0.0
0 0095	0 0091	0 0095	0 0094	0 0004	0.0083	0 0091	0.0085	0 0087	0.0088
177 9	214.2	179.2	262.8	243.3	69 3	278 4	279.5	172.4	0 1 8
0 85	1.00	96.0	1.14	1.15	98 0	1.14	1.06	0.93	0.87
0.91	0 85	60	0.89	0.82	0.81	08 0	000	: 0	160
-C7-3 5MO-1	-C7-3 5MO-2	-C7-3 SMO-3	-C7-3 5MO-4	N-C7-3.5MO-5	N-C7-8MO-1	-C7-6MO-2	-C7-6MO-3	-C7-6MO-4	CV-SMO.5

0 0 0 0 0

62 7

188.5

Notes:

+ Yald defined either as boal maxim um or as level region (zero slope) of load-slongation curve after initial rise.

+ Yald defined either as boal maxim um or as level region (zero slope) of load-slongation curve after initial rise.

- For all samples, rough leature may affect thickness measurements. Obtained as many as possible from clear places on film.

- Boals at criterio.

- I yald stress obtained from zero-slop e region.

- Valid stress obtained from local maximum.

Appendix B A Series Tensile Data

	P. Pose		•	•										•	•		•	• •	•	•	•	•		•		•	_	•	•				•	•	
Į de d	36		75.											10.4				*					2 2.				74.0								
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	*	876.0	813.0 826.7									693.3		0.19	659.7		632.7	8.44.3	728.0		1382.0		1346.0			992.0	964.3	956.0					1110.0	1040.0	
Bresk	Sed Dev		•										•	vo				•	,			•	•				-								•
Strength at	¥		126										;	•				100					¥ .				Z								•
	(kg/cm2)	123	128	:								=======================================	6 :	6 6		•	5	103	110		170		177	•		:	ss (904					197	•	P. 1
>	Section																																		
Strength at	Ş																																		
	(kg/cm2)																																		
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Al at Breek	(BB) 292 8	250.9	248.0								400		204.3			110.1	178.2	100.3	252.3	• • • • •	222 5	401.5	404.5	274.4	355.2		286.8	240.0					335.4	98	
	2.20	8 .	4 9 6 9 6 9 6								-		1.07	14.	1.78	1.78	1.40	0	2.22	•	4	3.32	3.30	2.31	2.33	6	2.20						2.76) i
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280 7 240 0 221 8 191 7 203 7	133 6 109.5 112.9 56.0 135.1	91.0 1114.9 10.0 6.0 4	50.8 132.0 74.5 140.8 72.8	200 100 110 100 100 100 100 100 100 100	169.0 112.6 178.0 178.7	2.0 24.7 137.8 95.2 121.0	140 0 123 6 92.3 15.5	128 2 61 4 23 6 6 0 84 4
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Notice:

- Yard defined either as local maxim em or as level region (zero alope) of load-elongation curve after initial rise.

- For at semples, rough learner may affect thickness measurements. For exposed samples, marine life deposits on filt-made it.

- For at semples, marine life deposits on filt-made as many as posed is from clear places on film.

- Breat at clamp

- Valed stress obtained from sore-level engine.

- Valed stress obtained from local maximum.

- Could not destinguish between breat ingland and yield load

Appendix C A Series Unexposed Tensile Data

	Load at Yield+ Load at Break		Al et Breek	Thickness.	Avg. Thickness	Tensile	Tensile Strength at Yield	Yield	Tensile	Stra	Break		% Elongation at Break	100	
Semple 10	(6 4)	<u>.</u>	Ê E	(E)	(E)	(RB/cm2)	9	\$0.000 0000	(Kg/cm2)	P	A 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	_	₹		
4-A1-0-1	•	2 47	363 2	0 0133					46 2			786.4			•
4-A1-0-2	•	2 62	387 7	0 0141					146 3			775 4			
N-A1-0-3		- 63	343.9	0 0123	0 0155				123.6	137 6	10.6	667 8	735.5	46.9	•
N-A1-0-4	•	3 25	3703	0 0235								740 6			•
N-A1-0-5	•	2 44	3436	0.0142					135.3			687 2			
		•				;			• • • • • • • • • • • • • • • • • • • •						
N-A2-0-1	6.0	2 55	434.6	96100		0 1						9 90			٥
4-A2-0-2	000	21.	532.5	0 0148		25.7	;				;	10650			۵
N-A2-0-3	1.04	9 40	532 4	0 0155	0 0151	25.0	51.7	-	/ 2/1	2 691		1064.8	1020 6	74.7	٥
N.A2-0-4	5	3.42	210.0	0 0158		50.3			* D/ I						ō.
4.A2.0.5	0.0	3.30	521.5	0.0153		0 OS			9 69 6			1043.0			۰
, 6. 6. 4. A	0 79	0 80	4.6.	0 0113		55.0			55.7			237 6			
	2	0 74	105.2	0 0113		54.4			51.6			210.4			} :
			2 2 2	0 0114	0.0116	52.5	54.0	6,1	5.19	97.0	5.3	373.0	278 5	77.	
			47.0	10.0		;	:					351.8		:	ì
				0000					63.0			9 01.0			
0.0.5	•	:													•
N-A4-0-1	1.32	1.06	149.4	0 0153		67.9			54.6			298.8			u
N-A4-0-2	5	1.4	240.	0 0164		67.5			6.68			461.6			v
N-A4-0-3	- 34	1 29	223.1	0 0162	0.0159	65.1	67.5	7.	62.7	62.4	•	446.2	408.9	0.00	
4.0.4	7 40	117	37.3	0.0162		68.0			96.9						
N.A4.0.5	- F	1.33	4.5	0.0152		6.89			69.0						
	3	•	i												,
N-AS-0-1	•	2.05	352.2	0.0138					117.0			704.4			•
N-A5-0-2	•	2.11	331.7	0.0138					120.4			663.4			
N-A5-0-3	٠	1 92	320 1	0.0135	0.0133				112.0	11.0	7.3	640.2	662.3	25.5	
N-AS-0-4	•			0 0126					108 1			644.2			
N-AS-0-5	•	1.63	320.4	0 0126					9.10			659.2			
	6		244.1	0 0000		36			55.3						4
			160	0 010 0		340			67.1			724.0			2 4
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N-A6-0-5	0	9.	288.2	0.0191		38.3			61.0			576.4			2
N-A7-0-1	•	0.84	94.5	0.0110					55.7			189.0			•
N-A7-0-2	•	0.83	61.5	0.0111					58.5						•
N-A7-0-3		1.07	103.4	0.0110	0.0114				5 02	4.0	-	206.8	197.7	34.3	•
N-A7-0-4	•	0 0 5	78.2	0 0113					5.6			156 4			•
N.A7.0.\$	•	0 65	1103	0.0110								238 6			•
7. O. 4	90	•	2610	0 0232		33.0			50 2						4
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N-A8-0-3			202 6	0.0225	0.0230	31.1	31 0	2 0	50.2	50.2	2.1	565.2	592.2	10.0	•
M-AB-0-4	0 65		286.8	0 0231		29 0			46.7			573 6			4
N-A8-0-5	80		3100	0.0230		30.8			51.2			620 0			Q.
									;						
N-A8-0-1	•	1 42	1612	0.0212					52.6			322 4			•
N-A9-0-2		1 26	187.4	0 0211								3748		1	•
N. A8-0-3	•	134	160 -	0 0210	0 0207				200	20 6	2.2	336.2	344 5	27.2	•
N-A8-0-4		1 37	1150	0 0203					9 7 G						•
N-A9-0-5	•	1 24	1332	0 0100					*						•

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+ Yield defend other as local maxim um or as level ingon (zero stope) of load-elongation curve after intel inte.

For all eartples, rough technic may affect thickness measurements for exposed autypies, marine life deposits on firm affects to obtain accurate thickness measurements. Obtained as many as possed to from clear places on firm.

6 Yield stress obtained from zero-elog o region.

6 Yield stress obtained from local maximum.

6 Maines growth at break point.

9 Could not designed from from from treak strength.

Appendix D

B Series Tensile Data

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184.6 23 24 25 27 28 29 29 29 29 29 29 29 29 29 29 29 29 29	184.8 23 23 24 25 25 25 26 27 29 29 29 29 29 20 20 20 20 20 20 20 20 20 20 20 20 20	•
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34 146 13 78.6 81.2 19.4 43 11.9 8 6.5 84.1 54.3 85.8 6.2 341.1 28.8 6.2 35.8 6.2 341.1 28.8 6.2 35.8 5.0 341.1 28.8 6.0 35.8 5.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 28.8 6.0 341.1 38.8	3.8	
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92 Ten 0.940 01 90	92 (.146 U.946 U.9	-
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Notes:

a - Break at Clamp

b - Could not desirguish between break ing load and yield load

o - Marrie Growth at Break

Appendix E Polypropylene/Polycaprolactone Tensile Data

	106.06	37.17	40.37	21.27	2	•	39.37
Awg	404.60	507.05		607.75	507.67	352.50	407.70
\$ 0.4 \$ 0.5 \$ 0.5 \$ 0.5	384.75 395.50	\$62.00 \$58.75 626.00 601.50	661.75 640.00 571.75 599.25	621.50 624.25 588.00 581.25 623.75	615 50 813.25 564.25	365 75 308.25	459.50 417.25 385.25 410.25 366.25
SEC Dev	10 02	6 6	75 21	8. 90.	94.23	2 8 69	11.10
P _V	203 37	308.74	335 20	309.28	336.03	227.45	208.63
(kg/cm2) 294 26	281 42 273.78 321.00 296 36	318 22 307 99 299 06 323 41 300.02	289.73 283.64 468.07 311.67 312.67	311.26 300.63 312.55 300.22 312.75	331.69 321.90 303.18 303.17 433.10	257.37 238.84 223.97 189.60	200.11 219.99 197.72 204.29 221.03
Sed Des	17.18	S 63	a) (a)	. 53	43.32		13.41
Avg	372.42	367.33	352.36	353.30	361.52	252 18	229.10
(kg/cm2) 362 68	370 67 370 67 386 26	374.77 358.41 366.75 369.61 367.12	348 08 344 21 355 38 355 10 356 02	356 94 346 32 347 28 361 74 354 22	330.49 367.36 350.82 325.84 433.10	252 96 259 61 246 91 250 89 250 55	243 62 242 88 213 58 221 95 223 93
(cm) 0 0054	0.0056	0.0060 0.0072 0.0064 0.0072	0.0045 0.0045 0.0045 0.0051	0.0074 0.0065 0.0068 0.0075	0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.0	0 00 54 0 00 60 0 00 60 0 00 60 0 00 58	0.0054 0.0062 0.0062 0.0062
(BB)	233.2 233.0 158.2	224 223 5 250 4 250 6 250 6	264.7 256.0 152.7 228.7 239.7	2 4 4 6 2 2 2 3 2 3 2 3 5 3 5 3 5 3 5 3 5 3 5 3	476.3 246.2 245.3 160.0	26.4 156.3 123.7 16.0 22.3	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
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Sample 10 N.PP/PCL-B1-1	N PP/PCL-81-2 N-PP/PCL-81-4 N-PP/PCL-81-4 N-PP/PCL-81-5	N-PP/PCL-82-1 N-PP/PCL-82-2 N-PP/PCL-82-3 N-PP/PCL-82-4	N-PP/PCL-83-1 N-PP/PCL-83-2 N-PP/PCL-83-4 N-PP/PCL-83-4	N.PP/PCL-84-1 N.PP/PCL-84-2 N.PP/PCL-84-3 N.PP/PCL-84-4 N.PP/PCL-84-4	N.PP/PCL-83-1 N.PP/PCL-83-2 N.PP/PCL-83-3 N.PP/PCL-83-4 N.PP/PCL-83-4	N-PP/PCL-86-1 N-PP/PCL-86-2 N-PP/PCL-86-3 N-PP/PCL-86-4 N-PP/PCL-86-4	A.PP/PCL-87-1 A.PP/PCL-87-2 A.PP/PCL-87-3 A.PP/PCL-87-3 A.PP/PCL-87-5

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